Continuous Counter-Current Extraction of a Dissolved Substance

From the Aqueous Phase by an Organic Solvent

by

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CONTINUOUS COUNTER CURRENT EXTRACTION OF A DISSOLVED
SUBSTANCE FROM THE WATER PHASE BY AN ORGANIC SOLVENT

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A number of mathematical formulas have been proposed hitherto relative to the extraction of a dissolved substance from an aqueous phase with an organic solvent [1-7]; graphical computation methods for extraction processes are also available [8-9].

However the major part of the proposed formulas and calculation methods has but limited applicability in practice because derivation of these formulas and the working-out of the calculation methods are effected, in most instances, without taking into account the specific characteristics of the prevalent technical extraction process conducted under industrial conditions.

The purpose of the present work was to determine the most advantageous conditions of a process for the extraction of acetic acid from the aqueous phase with an organic solvent, and to derive formulas for calculating the number of sections of the extractor, the concentration of acetic acid in the aqueous and the non-aqueous phase discharged from the extractor, depending upon the nature of the organic solvent and the ratio of the two phases.

The extraction process consists essentially in the procedure whereby an aqueous solution containing the acids to be extracted, for example, acetic, formic, propionic and oleic acid,

- 1 -

is treated with a given organic solvent, the acetic acid thus being distributed between water and solvent in proportions determined by the distribution coefficient of the acid in the two liquid phases, and by the quantitative ratio of these phases.

Transfer of acid from the aqueous into the non-aqueous phase takes place until a state of equilibrium is reached which is determined by the distribution coefficient

$$K = \frac{x}{y}$$

(1)

where x is the concentration of the dissolved substance in the organic solvent, and y its concentration in water.

The distribution law expressed by formula (1) holds only in the case where the dissolved substance does not interact chemically with either solvent medium and where its molecular state is the same in both solvent media. In addition the solutions must be of low concentration. The solvent media must display a low mutual solubility; in any event their mutual solubility must change but little with alterations of the concentration of the dissolved substance.

If the substance being extracted forms bimolecules in the organic solvent, and monomolecules in water, i.e., where the molecular weight of such a substance is twice as large in the organic solvent as it is in water, then the distribution coefficient is given by the formula

- 2 -

$$K = \frac{x}{y^2} \qquad (2)$$

If the dissolved substance dissociates into ions, and its degree of dissociation in the organic solvent is α_1 , and in water α_2 then the distribution law assumes the following form:

$$K = \frac{x(1-\alpha_1)}{y(1-\alpha_2)} \qquad (3)$$

wherein X and Y are total concentrations. While $(1-\alpha_1)$ and $(1-\alpha_2)$ are the concentrations of the undissociated portion of the acid being extracted, in the organic solvent and in water, respectively.

If dissociation occurs only in water then the distribution law becomes:

$$K = \frac{x}{y(1-\alpha_2)} \qquad (4)$$

From the distribution coefficient equation (3) it follows, that the presence of associated molecules in the solvent has a beneficial effect on the course of the extraction. Since an extract is obtained containing a higher concentration of acetic acid. If the "acid water" has dissolved in it several acids, a different distribution coefficient corresponds to each of them

- 3 -

$$K_1 = \frac{x_1}{y_1} \; ; \; K_2 = \frac{x_2}{y_2} \; ; \; K_3 = \frac{x_3}{y_3} \; \dots \text{ etc.} \quad (5)$$

The extraction process can be performed by means of
equipment that operates intermittently or continuously. On
intermittent extraction with fresh batches of solvent, the de-
crease of acetic acid concentration in the processed water to a
desired degree of exhaustive extraction (0.1 - 0.2 percent) al-
ways leads to the production of extracts containing insigni-
ficant concentration of acetic acid even when the solvent is
divided into a considerable number of aliquot portions.

In due order to obviate the use in the extraction of
large amounts of solvent and to remove completely from the water
the substances dissolved therein, it is indispensible to con-
duct the extraction process in a continuously operating system
utilizing the counter current principle.

Feed and discharge of aqueous solution and solvent from
the extractor depend on the specific gravity of the solvent.
If the specific gravity of the solvent is less than one, the
aqueous solution is introduced into the extractor from the top,
and the solvent from the bottom; if the specific gravity of the
solvent is greater than one, then the solvent is introduced at the
top and the aqueous solution at the bottom. Flow of the medium
being extracted and of the extractant through the extractor in
counter current course ensures first of all the highest concen-
tration of acetic acid in the extract, since the solvent

-4-

on passing through the extractor gradually becomes saturated with acetic acid, and prior to leaving the extractor comes in contact with fresh "acid water" which contains the highest concentration of acetic acid; and secondly, the fresh solvent containing no acetic acid, comes in contact with "acid water" of least concentration, as a result of which a more complete removal of acetic acid from the "acid water" is attained.

It must be kept in mind that the phenomena occurring in the extractant medium and in that being subjected to extraction are rendered more complex by the changes in mutual volubilities of these liquids which take place on alteration of the concentration in the liquids of the substance being removed by extraction. Hence, generally speaking, the distribution of, for example, acetic acid between water and solvent cannot be expressed by the constant proportion of the concentrations of the acetic acid in the two dissolving media within each section of the extractor. In first approximation, however, we can assume that: (1) The composition of the solvent and of the "acid water" undergoes continuous change only with respect to their content in acetic acid; (2) in each section there is reached an equilibrium in the distribution of the acid between water and solvent; (3) the components of the "acid water" comprise only acetic acid, and, (4) the molecules of acetic acid in the water as well as in the organic solvent are neither dissociated nor associated and the coefficient of distribution conforms to the simple formula:

$$K = \frac{x}{y} \tag{6}$$

- 5 -

Let W liters of water containing Z grams of acetic acid be introduced into the extractor with each liter of "acid water".

Also, let L liters of organic solvent containing X_0 grams of acid per one liter of pure solvent, be introduced into the extractor. When pure solvent is being introduced into the extractor $X_0 = 0$.

Let us denote the content of acetic acid in sections 1, 2, 3...n (respectively): in the water by y_1, y_2... y_n and in the solvent by x_1, x_2, x_3...x_n.
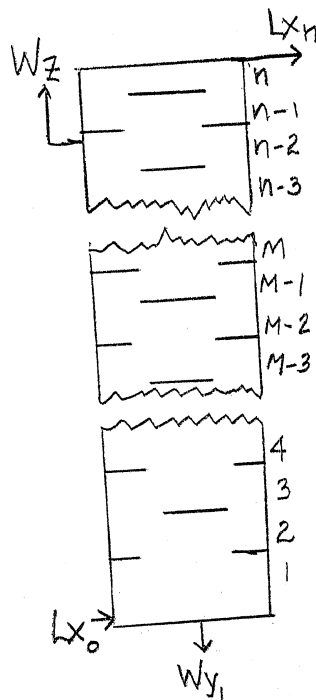
The sections are numbered beginning at the bottom (Figure 1)

Let us assume that within each section there is reached an equilibrium of acetic acid in the water and the solvent.

Material balance with respect to acetic acid within each section will then be as follows:

Intake of acid. (1) The amount of acid (in grams) being introduced into the first section of the extractor, with L liters of solvent is equal to Lx_0. (2) The amount of acid (in grams) flowing out of two sections into the first [section] with W liters of water is equal to Wy_2; total intake of acid $Lx_0 + Wy_2$ grams.

Expenditure of acid. (1) The amount of acid (in grams) leaving the extractor with W liters of water is equal to Wy_1; (2) The amount of

Figure 1.

- 6 -

acid (in grams) carried from the first section into the second with L liters of solvent is equal to Lx_1; total expenditure of acid $Wy_1 + Lx_1$ grams.

On comparing intake with expenditure we have:

$Lx_0 + Wy_2 = Wy_1 + Lx_1$.

Assuming Xo to equal 0, we also have Lx_0 equal to 0.

Using the coefficient of distribution $K = \dfrac{x}{y}$, let us express the variable x as a function of y, that is, $x = K_1 y$. Then,

$$y_2 - y_1 = \frac{Lx_1}{W} = \frac{LK_1}{W} y_1$$

In an analogous manner we have for the second, third etc. section

$$y_n - y_{n-1} = \frac{LK_1}{W}\left(y_{n-1} - y_{n-2}\right) \qquad (7)$$

By the same procedure we find the expression giving the difference of concentration of acetic acid in the feed liquid and the concentration of acid in the aqueous layer within the last section

$$z - y_n = \frac{LK_1}{W}\left(y_n - y_{n-1}\right) \qquad (7a)$$

In each of the equations so obtained the ratios

$$\frac{y_2 - y_1}{y_1} \;;\quad \frac{y_3 - y_2}{y_2 - y_1} \;;\quad \frac{y_4 - y_3}{y_3 - y_2} \qquad \text{etc are constant quantities}$$

equal to $\dfrac{LK_1}{W}$.

- 7 -

Substituting in equations (7), beginning with the second equation ($y_2 - y_1$), its expression taken from the first equation, in the third equation ($y_3 - y_2$) its expression taken from the second equation, etc, we have

$$y_2 - y_1 = \frac{LK_1}{W} y_1, \cdots \ y_n - y_{n-1} = \left(\frac{LK_1}{W}\right)^{n-1} \cdot y_1$$

(8)

Adding equations (8) we obtain:

$$y_n - y_1 = \frac{LK_1}{W} \left[\frac{\left(\frac{LK_1}{W}\right)^{n-1} - 1}{\frac{LK_1}{W} - 1} \right] y_1$$

denoting $\frac{LK_1}{W} = p$ and designating it thereafter as the parameter we have:

$$y_n = y_1 \ \frac{p^n - 1}{p - 1}$$

(9)

Then from formula (7a) and the last equation (8), the content of acetic acid in the aqueous phase introduced into the extractor, will be equal to:

- 8 -

$$Z = y_n + p^n \cdot y_1 \qquad (10)$$

Substituting in formula (10) in place of y_n its value from formula (9) we have

$$Z = y_1 \frac{p^{n+1} - 1}{p - 1} \qquad (11)$$

Using formulas (9) and (11), we can determine the concentration of acid in the aqueous phase of the first and the last section, depending upon the number of sections in the extractor and the concentration of acid in the feed liquid, by means of formulas

$$y_1 = Z \frac{p - 1}{p^{n+1} - 1} \qquad (12)$$

$$y_n = Z \frac{p^n - 1}{p^{n+1} - 1} \qquad (13)$$

Here, three possible cases may take place: (1) when $p < 1$; (2) when $p = 1$; (3) when $p > 1$.

Let us consider all three of the above-indicated possibilities. Eliminating n in equations (12) and (13) we have:

- 9 -

$$y_1 + Py_n = z \qquad (14)$$

Let $p \neq 1$, then from (12):

$$\frac{dy_1}{dn} = \frac{-z(p-1)p^{n+1}\ln p}{(p^{n+1}-1)^2};$$

$$\frac{d^2 y_1}{dn^2} = \frac{z(p-1)p^{n+1}(p^{n+1}+1)\ln^2 p}{(p^{n+1}-1)^3} \qquad (15)$$

From formula (15) it follows that:

$$\frac{dy_1}{dn} < 0, \quad \frac{d^2 y_1}{dn^2} > 0$$

Within the interval $0 < n < +\infty$ the acid con-
centration y_1 in the processed acid water decreases with increasing
n. The curve (12) is concave upwards. With $p < 1$ it has an
asymptote y = z (1-p).

Thus within the interval $0 < n < +\infty$ with $p < 1$ we
have the double inequality

$$z > y_1 > z (1-p) \qquad (16)$$

On the basis of the inequality (16) a preliminary estimate
can be given of a process of extraction of volatile organic acids

- 10 -

from an aqueous phase with a given organic solvent when $p < 1$

Using inequality (16) and knowing the concentration of the acid in the aqueous phase fed into the extractor, it is possible to determine forthwith whether it would be advantageous, from an economic standpoint, to affect the removal of acid from the aqueous phase with the organic solvent.

If, on the other hand, $p > 1$, then y_1, is determined by means of formula (12), decreases, having zero as the limit.

From equation (14) we have

$$\frac{dy_n}{dn} > 0, \quad \frac{d^2 y_n}{dn^2} < 0$$

Within the interval $0 < n < +\infty$, y_n is a rising function. The curve (13) is concave downwards.

From formula (14) it follows that when $p < 1$ curve (13) has an asymptote $y = z$, and when $p > 1$, an asymptote $y = \frac{z}{p}$.

Let us now consider the case when $p = 1$.

From equations (12) and (13) it follows that the concentration of the acid in the aqueous phase being discharged from the extractor can be determined by means of the formula: $y_1 = \frac{z}{n+1}$, and the concentration of the acid in the same phase within the section n, by means of the formula:

$$y_n = \frac{nz}{n+1} \; .$$

Let us now determine the rule which governs the change of acid concentration in the different sections of the extractor. To

- 11 -

do so let us consider the difference of acid concentration in the
aqueous layer of two consecutive sections.

On the basis of the material balance we have in each section
of the extractor:

$$y_2 - y_1 = p y_1, \ldots, y_M - y_{M-1} = p^{m-1} \cdot y_1 \qquad (17)$$

From a series of these equations it is apparent that with a
constant y_1, - acid concentration in the aqueous phase following
out of the first section of the extractor, the difference of con-
centrations of acid in the water will increase. When $p > 1$, and
decreases when $p < 1$, in geometric progression.

Adding equations (17), we find the concentration of acid in
the aqueous phase of the m - th section:

$$y_M = \frac{y_1 \left(p^m - 1 \right)}{p - 1} \qquad (18)$$

Substituting for y_1 its value given by equation (12), we have:

$$y_M = \frac{z \left(p^m - 1 \right)}{p^{n+1} - 1} \qquad (19)$$

In the case when p=1, it follows from formula (18) that:

$$y_M = M y_1 \qquad (20)$$

- 12 -

From formula (20) it follows that the acid concentration in the aqueous layer increases in arithmetical progression. When the concentration of acid in the water flowing out of the first section remains constant.

The question arises: which of the three instances considered above with respect to the value of p is the most advantageous for a given number of sections of the extractor, or, more precisely, in which of the three instances is the acid concentration in the water discharged from the extractor the lowest?

If for the given number of sections, we designate the acid concentration in the aqueous phase of the first section by: y_{11} when $p_1 < 1$; by y_{12} when $p_2 = 1$, and by y_{13} when $p_3 > 1$, it can be shown that when $n > o$ the concentration of acid in the aqueous layer of the first section is greater in the case when $p = 1$, than in the case when $p > 1$ and is less than in the case when $p < 1$; that is, we have the following inequality:

$$ y_{13} < y_{12} < y_{11} \qquad (21) $$

To prove the existence of this inequality let us consider the exponential function $y = x^{n+1} = f(x)$, where n is a whole positive number.

Let the argument x have two given values:
$$ x_1 = 1, \quad x_2 = p_3 \text{; } p_3 > 1. $$

According to the Lagrange formula of terminal increase of a function

- 13 -

$$f(x_2) - f(x_1) = (x_2 - x_1) f'(x_0); \quad x_1 < x_0 < x_2,$$

we have:

$$p_3^{n+1} - 1 = (p_3 - 1)(n+1) x_0^n; \quad x_0 = 1 + \theta_1(p_3 - 1)$$

from which:

$$\frac{p_3^{n+1} - 1}{p_3 - 1} = (n+1) x_0^n$$

since $x_0^n > 1$, then $\dfrac{p_3^{n+1} - 1}{p_3 - 1} > n+1$ if $\dfrac{p_3 - 1}{p_3^{n+1} - 1} < \dfrac{1}{n+1}$

Multiplying the latter expression by an arbitrary positive number z, we have:

$$\frac{z(p_3 - 1)}{p_3^{n+1} - 1} < \frac{z}{n+1}$$

$$(22)$$

Now, let argument x of the same exponential function be given the value $x_1 = p_1$; $x_2 = 1$; $o < p_1 < 1$.

Using the Lagrange formula we have

$$1 - p_1^{n+1} = (1 - p_1)(n+1) x_0^n$$

where

$$x_0 = p_1 + \theta(1 - p_1); \quad 0 < \theta < 1.$$

- 14 -

$$\frac{1-p_1^{n+1}}{1-p_1} = (n+1)x_o^n; \; x_o^n < 1, \; \frac{1-p_1^{n+1}}{1-p_1} < n+1$$

hence,

$$\frac{1-p_1}{1-p_1^{n+1}} > \frac{1}{n+1}$$

Multiplying both members of this inequation by the same arbitrary positive number z, we have:

$$\frac{z(1-p_1)}{1-p_1^{n+1}} > \frac{z}{n+1} \tag{23}$$

Fron inequalities (22) and (23), which contain the expressions of acid concentration in the aqueous layer of the first section, we find for $n > 0$;

$$Y_{13} < Y_{12} < Y_{11} \tag{23a}$$

The change of concentration of acid in the water discharged from the extractor, as a function of the number of extractor sections, for all cases is shown in figure 2.

It is possible to show the existence of another inequality, namely

$$Y_{n3} < Y_{n2} < Y_{n1}$$

Where yn_3, yn_2 and yn, is the concentration of acid in the last -- upper section. This inequality shows that the concentration in the last section, for the same value of n, in the case where $p3 > 1$ will be lower than in the case when $p_2 = 1$, and that the

- 15 -

concentration in the last section when $p_2 = 1$, will be lower
than that which obtains when $p_1 < 1$.

Let us consider the two functions:

$f(x) = x^n - 1$, $\phi(x) = x^{n+1} - 1$. Where n is a whole
positive number ($n > 0$), which determines the number of extractor
sections.

Let argument x be given the values:

$$x_1 = p_1, \quad x_2 = 1; \quad o < p_1 < 1$$

According to the formula of Cauchy:

$$\frac{f(x_2) - f(x_1)}{\phi(x_2) - \phi(x_1)} = \frac{f^1(xo)}{\phi'(x_0)} \quad ,$$

where $p_1 \quad x_0 \quad 1$, we have:

$$\frac{1 - p_1^n}{1 - p_2^{n+1}} = \frac{n}{(n+1) x_0}$$

Since $x_0 < 1$ we have $\dfrac{1 - p_1^n}{1 - p_1^{n+1}} > \dfrac{n}{n+1}$

If we take the same functions for the values $x_1 = 1$ and
$x_2 = p_3$ where $p_3 > 1$ and using the same formula of Cauchy, we have:

$$\frac{p_3^n - 1}{p_3^{n+1} - 1} = \frac{n}{(n+1) x_0}, \text{ where } 1 < x_0 < p_3$$

Since now $x_0 > 1$, we have $\dfrac{p_3^n - 1}{p_3^{n+1} - 1} < \dfrac{n}{n + 1}$

Thus,

-16-

$$\frac{P_3^{n-1}}{P_3^{n+1}-1} < \frac{n}{n+1} < \frac{1-P_1^n}{1-P_1^{n+1}}$$

(24)

If inequations (24) are multiplied by the positive number

z, we have the expression for the concentration in the last section:

$$y_{n3} < y_{n2} < y_{11}$$

(25)

Figure 3 shows the graphs of all three functions. Herein
before we have obtained the double inequality:

$$y_{13} < y_{12} < y_{11}$$

(26)

Wherein y_{13} is the concentration of acetic acid, in the water
discharged from the extractor, when $p_3 > 1$; y_{12} is that when $p_2 = 1$;
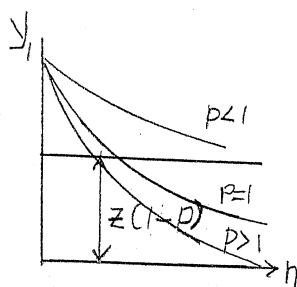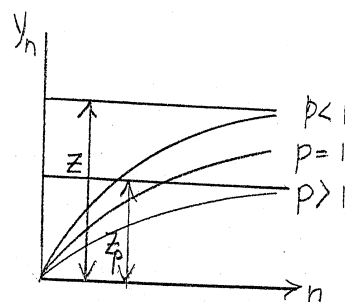and y_{11} when $p_1 < 1$.

Figure 2

Figure 3

-17-

The double inequality (26) makes it possible readily to prove another double inequality:

$$n_3 < n_2 < n_1 \tag{27}$$

Let y_1 and z (concentration of acetic acid in the feed liquid) have the same values for all three instances: $p_3 > 1$; $p_2 = 1$: $p_1 < 1$.

$$y_1 = \frac{z(p_3 - 1)}{p_3^{n_3+1} - 1} = \frac{z}{n_2 + 1} = \frac{z(1 - p_1)}{1 - p^{n_1+1}} \tag{28}$$

wherein n_3 - if the number of sections in the extractor, when $p_3 > 1$, n_2 - the number of sections when $p_2 = 1$ and n_1 the number of sections in the extractor when $p_1 < 1$.

From equations (28) it follows that:

$$\frac{p_3 - 1}{p_3^{n_3} - 1} = \frac{1}{n_2 + 1} , \quad \frac{1}{n_2 + 1} = \frac{1 - p_1}{1 - p_1^{n+1}} \tag{29}$$

But in the case of any number of sections in the extractor, from inequalities (23a) we have:

$$\frac{z(p_3 - 1)}{p_3^{n_3+1} - 1} < \frac{z}{n_3 + 1} , \quad \frac{z}{n_3 + 1} < \frac{z(1 - p_1)}{1 - p_1^{n+1}} \tag{30}$$

using equations (29) we obtain from inequalities (30)

-18-

$$\frac{1}{n_2+1} < \frac{1}{n_3+1} \; ; \; \frac{1}{n_1+1} < \frac{1}{n_2+1} \tag{31}$$

From inequality (31) we Have:

$$n_3 < n_2 < n_1 \tag{32}$$

The existence of the double inequality (32) presupposes that:

$$y_1 > z \; (1 - p_1).$$

This follows from formula:

$$y_1 = \frac{z \; (1 - p_1)}{1 - p_1^{n_1+1}} \qquad \text{when } o < P_1 < 1.$$

Let us now examine the change of the number of sections (n) depending upon the change of parameter p. Let us consider the case when $p > 1$ and $o < p < 1$.

From formula $y_1 = z \left(\dfrac{1 - p}{1 - p^{n+1}} \right)$ it follows that:

$$p^{n+1} = \frac{z(p-1)}{y_1} + 1 \tag{33}$$

From (33) we find the expression for the derivative:

$$\frac{dn}{dp} = - \; \frac{np^{n+1} - (n+1)p^n + 1}{(p-1)p^{n+1} \ln p} \tag{34}$$

- 19 -

In the equation (34) the denominator $\dfrac{dn}{dp}$ is positive. Consider the numerator $y_o = np^{n+1} - (n+1)\, p^n + 1$, where n is a whole positive number different from zero.

y_o can be written in the following form:

$$y_o = (p-1)^2 \ [np^{n-1} + (n-1)\, p^{n-2} + \ldots + 1]$$

From this it follows that:

$$y_o > 0 \tag{35}$$

Thus, when $o < p < 1$ we have $p > 1$ $\dfrac{dn}{dp} < o$

With increasing value of the parameter p the number of sections in the extractor decreases.

Let us now assume that in formula (12)

$$y_1 = \frac{z\,(p-1)}{p^{n+1} - 1}$$

z and n = constant values. From formula (12) we have:

$$y'_{1p} = -\frac{z\,[np^{n+1} - (n+1)\, p^n + 1]}{(p^n - 1)^2} \tag{36}$$

As was previously demonstrated (35) : $np^{n+1} - (n+1)\, p^n + 1 > o$ with $n > o$ in both instances $p > 1$, $o < p < 1$. From (36) it follows that; $y^1_{1p} < o$.

With increasing parameter p, y_1 decreases, when $p > 1$ as well as when $o < p < 1$.

-20-

Let us now examine the change of concentration of acetic acid in the last section.

$$y_n = Z \frac{p^n - 1}{p^{n+1} - 1} \tag{37}$$

From formula (37):

$$y'_{n_p} = - \frac{Z p^{n-1} \left[p^{n+1} - (n+1) p + n \right]}{(p^{n+1} - 1)^2} \tag{38}$$

The polynominal $y_0 = p^{n+1} - (n+1) p^n + n$ can be written in the following form:

$$y_0 = (p-1)^2 (p^{n-1} + 2p^{n-2} + 3p^{n-3} + \ldots + n)$$

Hence, when $p > o$ we have $y_0 > o$. Thus $y^I_{np} < o$.

With increasing p, yn decreases in both instances: $o < p < 1$ and $p > 1$.

CONCLUSION

1. It was shown that at the same concentration of feed liquid the concentration in the discharged water and the concentration in the extract, with a given number of sections, depends upon the number p, for which there have been established three characteristic cases $o < p < 1$; $p = 1$; $p > 1$.

2. In all three cases there was found a correlation between the value of p, the number of sections, concentration of

acid in the feed and in the discharged water, and the concentration in the extract.

3. It has been demonstrated that for a given number of sections in the extractor and concentration of acid in the feed liquid the lowest concentration in the discharged water corresponds to the case when $p > 1$.

4. When $p < 1$ concentration of acid in the extractor will always satisfy the inequa[li]ty $X_n < k \frac{z}{p}$.

5. When $p < 1$ concentration in the discharged water will be $y_1 > z \bigcirc (1-p)$.

6. For the acid concentration in the aqueous layer of the first section, with the number of sections being the same, the existence _of the_/following inequa[li]ty was demonstrated: $y_{13} < y_{12} < y_{11}$. y_{13} - concentration of acetic acid in the discharged water when $p > 1$; y_{12} - concentration of acetic acid in the discharged water when $p=1$; y_{11} - concentration of acetic acid in the discharged water when $p < 1$.

7. The existence of a double inequa[li]ty was demonstrated: $n_3 < n_2 < n_1$, where n_3 is the number of sections when $p > 1$; n_2 - the number of sections when $p=1$; n_1 - the number of sections when $p < 1$ with given values of z and y_1 - the same in all three of the instances indicated.

8. It has been demonstrated that with increasing value of p the number of sections (n) in the extractor decreases when $p < 1$ and $p > 1$.

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